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(COMPATIBILITY OF VAPOR DEPOSITED B, SiC, AND TiB₂ FILAMENTS WITH SEVERAL TITANIUM MATRICES)

JAMES A. SNIDE
CAPTAIN, USAF

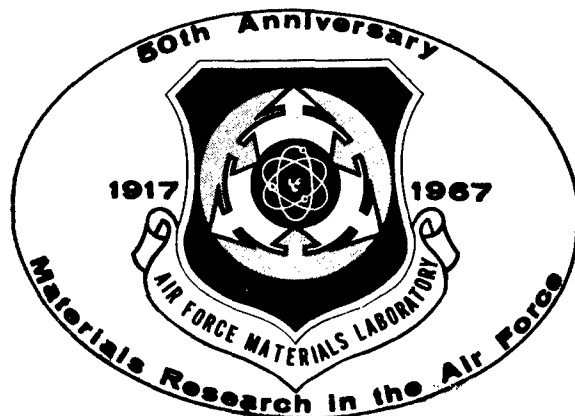
TECHNICAL REPORT AFML-TR-67-354

FEBRUARY 1968

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**COMPATIBILITY OF VAPOR DEPOSITED B, SiC, AND TiB₂
FILAMENTS WITH SEVERAL TITANIUM MATRICES**

*JAMES A. SNIDE
CAPTAIN, USAF*

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FOREWORD

This report was initiated in the Air Force Materials Laboratory under Project No. 7353, "Characterization of Solid Phase and Interface Phenomena in Crystalline Substances", Task No. 73530606, "Fundamentals of Composites." It was released by the author 1 October 1967 for publication as a technical report.

This report was authored by Capt. James A. Snide of the Advanced Metallurgical Studies Branch, Metals and Ceramics Division, Air Force Materials Laboratory, Research and Technology Division, Wright-Patterson Air Force Base, Ohio. It covers a study conducted from September 1965 to October 1967.

The author would like to acknowledge Dr. K. Richards of Kennecott Copper Corporation, Salt Lake City, Utah (formerly with the Aerospace Research Laboratories at WPAFB, Ohio) for his help and encouragement in the planning and conduct of the experimental effort described in this report. This work was initially started as a joint effort with Dr. Richards prior to his leaving ARL.

The author would like to acknowledge the contributions of the following people to the effort: Mr. C. R. Underwood and Mr. E. H. Wooley of the AFML in the preparation of metallographic specimens; Mr. L. W. Whittaker of the University of Dayton under Air Force Contract AF 33615-67-C-1187 for the preparation of diffusion couples; Mr. L. Bates of Systems Research under Air Force Contract AF 33615-67-C-1421 for the electromicroscopy; and Mr. M. Rosenblum of the University of Cincinnati under Air Force Contract AF 33615-67-C-1178 for the microprobe work.

This technical report has been reviewed and is approved.



C. T. LYNCH
Chief, Advanced Metallurgical
Studies Branch
Metals and Ceramics Division
Air Force Materials Laboratory

ABSTRACT

Start 1
Silicides of B, SiC, and TiB₂ vapor deposited on tungsten wire
 The filament-matrix compatibility was evaluated for B, SiC, and TiB₂ vapor deposited filaments with high purity, commercial purity titanium, and Ti-6Al-4V. Filament-matrix diffusion sandwiches were prepared by solid state resistance bonding. These samples were subsequently heated at 800-991°C for times of 1-100 hours. The reaction rates and reaction products were evaluated using light microscopy, electron replicas, microhardness measurements, and microprobe analysis. *Ja P 3*

comp 3.0
 The order of decreasing filament-matrix interaction was B, SiC, and TiB₂ in each of the three materials. High purity Ti, commercial purity Ti, and Ti-6Al-4V is the order of decreasing reactivity with the filaments after 100 hours at 850-991°C. The reaction of B with unalloyed titanium is characterized as the formation of a TiB₂ layer adjacent to the filament with an external acicular TiB layer. The reaction layers were formed by the predominately outward diffusion of the boron as evidenced by the lack of recession of the initial filament diameter and the void formation in the filament. The interaction of B with Ti-6Al-4V is characterized by the formation of TiB₂ with the rejection of Al ahead of the advancing TiB₂ front which stabilizes the α -titanium phase around the TiB₂ reaction layer. The SiC filament reacts with titanium to form a multi-phased reaction layer. The SiC-Ti interaction takes place by the simultaneous growth inward and outward of the reaction zone. A uniform distribution of 0.1 weight percent titanium was observed in the SiC filament after 100 hours at 850°C. The TiB₂ filament reacted with titanium to form a TiB layer adjacent to the filament. The SiC and TiB₂ filaments did not react with their respective tungsten or molybdenum core after 100 hours at 850°C. *The inner concentric ring shown in the electron micrograph of the TiB₂ filament was a result of the deposition process and not TiB₂ filament - high purity titanium matrix interaction.*

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SECTION I

INTRODUCTION

The technical potential of metal matrix composites has been recently reviewed (Reference 1). In this article, it was pointed out that the major problem in developing metal matrix composites for elevated temperature use is the lack of filament-matrix compatibility. At the Air Force Materials Laboratory, various filaments have been screened to determine their compatibility with the more important aerospace metals. Figures 1 through 3 show the interaction of B, SiC, and TiB_2 filaments with iron, nickel, and cobalt, after anneals for various times at 900°C. Since these metals are the basis of the superalloys, 900°C was selected as the screening temperature as this is a representative use-temperature for the superalloys. This initial screening showed that the reaction of B, SiC, and TiB_2 filaments is sufficiently rapid with iron, nickel, and cobalt, at 900°C that these filaments cannot be used to reinforce the superalloys without some approach to improve the filament-matrix compatibility.

Similar studies were started to evaluate the interaction of these filaments for use in titanium matrices. Titanium is an extremely important aerospace metal which would receive wider use if the strength and stiffness could be enhanced by reinforcing with low density, high modulus filaments. The purpose of this investigation was to study these filament-matrix interactions to determine the rate of interaction and to assess the potential of reinforcing these matrices with B, SiC, and TiB_2 filaments.

SECTION II

EXPERIMENTAL

1. MATERIALS

Table I lists some properties of the various filaments which were used in this investigation. Boron and SiC filaments are production filaments which are deposited on a 1/2 mil tungsten wire substrate. The TiB_2 is a filament which is in the early stages of development. This filament is deposited on a 1 mil molybdenum substrate.

Table II lists the major impurities of the three types of titanium materials. The iodide titanium listed is a three-pass, zone-refined 1/4 inch rod. The commercial purity titanium listed is Grade 55 1/16 inch sheet. The titanium alloy (Ti-6Al-4V) listed is commercially procured 1/16 inch sheet.

2. PROCEDURE

Small composite specimens for this compatibility study were prepared by solid state, electrical resistance bonding of two identical metal sheets with filaments placed between them. Figure 4 shows a schematic of the specimen lay-up which was used. The steel wires were used both to complete the electrical circuit and to prevent the filaments from being crushed before the metal coupons reached the bonding temperature. Prior to bonding, the metal couples were metallographically polished through 600 grit paper to remove any surface layer and to prepare a smooth surface. After polishing, the samples were rinsed consecutively in acetone, methyl alcohol, and distilled water. The filaments were similarly cleaned prior to bonding. The specimens were bonded in an argon atmosphere with low voltage, high amperage current. In this work, the welding temperature was not measured, but was roughly estimated at approximately 900°C by visual observation of the radiating sample. Pressure is applied while the sample is at temperature and visible deformation occurred which insured good metal flow around the filaments. Bonding times were of the order of 5 to 10 seconds. When the current was turned off, the specimen cooled in flowing argon to room temperature in approximately 45 seconds.

After bonding, the samples were polished to show the filaments in cross section to determine the extent of the filament-matrix interaction which occurred during the bonding operation. Next, the filament-matrix sandwich was removed from the Bakelite mount and prepared for the thermal treatment. The samples were wrapped loosely in tantalum foil and sealed in an evacuated ($<10^{-6}$ Torr) quartz ampule to prevent oxidation of the titanium during the thermal treatment.

A series of samples were annealed for 100 hours at 850°C. Selected samples were annealed for times of 1, 10, and 100 hours at selected temperatures. Metallographic samples were prepared after each thermal treatment to determine the extent of the filament-matrix interaction. The titanium matrix was etched with a solution of 2 ml HF, 4 ml HNO_3 , and 94 ml H_2O . The filament-matrix interaction was evaluated using microhardness traverses, single-stage electron microscope replicas, conventional metallography, and the electron microprobe.

SECTION III

RESULTS AND DISCUSSION

1. HIGH PURITY TITANIUM

Figure 5 shows the interaction of the B, SiC, and TiB₂ filaments with high purity titanium after a 100 hour isothermal anneal at 850°C. If the size of the reaction layer between the matrix and the filaments is taken as the measure of the degree of interaction, B, SiC, and TiB₂ is the order of decreasing filament-matrix interaction. As it has been shown in previous studies conducted at this laboratory the reaction can be represented by a parabolic equation (Reference 2). Figure 6 shows a comparison of the parabolic rate constants for the equation

$$x^2 = kt$$

(1)

where x is the reaction zone width in centimeters, t is time in seconds, and k is the parabolic rate constant. The parabolic rate constants shown in Figure 6 were calculated by using only the reaction zone thickness after 100 hours.

a. Boron Filament

Figure 7 shows the typical reaction between boron and high purity titanium after a 100 hour anneal at 850°C. The reaction layer adjacent to the boron is TiB₂. Figure 8 is an electron microscope replica of the etched surface of the polished sample which shows the 14μ TiB₂ layer. This TiB₂ phase was identified by Blackburn et al (Reference 2) in their investigation of the interaction of boron with Ti-8Al-1Mo-1V. The acicular structure shown in Figure 7 ahead of the TiB₂ is probably TiB. The TiB phase was indicated by results from the electron microprobe which showed a decrease in boron concentration in comparison to the TiB₂ phase. These reaction layers were formed by the predominately outward diffusion of the boron as evidenced by the lack of recession of the initial filament diameter and the void formation in the filament. The presence of the TiB₂ and TiB phases agree with the titanium-boron phase diagram (Reference 3).

Figure 9 shows the core of the boron filament after 100 hours at 850°C. The tungsten has been completely converted to the respective borides as previously reported by Hammond et al (Reference 4) as there is no unreacted tungsten present. The conchoidal fractures overlap around the core. These fractures probably occur during polishing and are the result of the complex residual stress state of the filament because of the tungsten-boron interaction during the filament manufacture (Reference 4). Figure 10 shows the conchoidal fractures in the boron filament which appears as gross void in the filament in the light micrograph (Figure 7). These fractures occur during polishing and probably are initiated at voids caused by the condensation of vacancies which result from the predominantly outward diffusion during the interaction with titanium.

b. Silicon Carbide Filament

Figure 11 shows the interaction between a SiC filament with high purity titanium after 100 hours at 850°C. The reaction layer is multi-layered which agrees with the phase diagram (Reference 5) which indicates that TiC, Ti₅Si₃, and TiSi₂ should form between diffusion

couple of titanium and SiC. Microprobe work showed a uniform distribution of approximately 0.1 weight percent of titanium in the SiC filament. This is in agreement with the earlier findings of Feingold (Reference 6). The residual titanium-to-titanium bond line is present although some grain growth has occurred across this interface. Figure 12 shows more clearly the structure of the reaction layer. The initial SiC diameter (prior to the thermal treatment) coincides with the scalloped looking phase; therefore, the SiC-titanium interaction proceeds by a simultaneous recession of the SiC diameter and growth of the reaction zone into the titanium. The coherent nature of the SiC filament, in comparison with the B filament after interaction, can probably be attributed to this simultaneous inward and outward growth of the reaction layer. Figure 13 shows that the reaction zone between the tungsten core and the SiC is less than 0.25μ ; therefore, the majority of the core is unreacted tungsten. Adler et al (Reference 7) has shown that the tungsten core starts to react rapidly with the SiC at 1000°C .

c. Titanium Diboride Filament

Figure 14 shows the interaction of the TiB_2 filaments with high purity titanium to form a reaction layer adjacent to the filaments which is probably a TiB phase. The TiB phase was indicated by the electron microprobe results which showed a decrease in boron concentration in comparison to the TiB_2 phase. Figure 15 shows more clearly the structure of filament and the filament-matrix interaction. No interaction can be seen between the molybdenum core and the TiB_2 filament. The inner concentric rings are present in the as-deposited filament and are a result of the deposition process and not filament-matrix interaction. The TiB phase is approximately 6μ in width. Figure 16 shows the increase in the matrix hardness from 230 to DPH 777 as a result of the boron diffusion into the matrix. The triangular areas adjacent to the filament are matrix void caused by insufficient matrix deformation during sample bonding. The hardness of molybdenum does not change as a result of the thermal treatment as the molybdenum core does not react with TiB_2 to any detectable amount after 100 hours at 900°C .

2. COMMERCIAL PURITY TITANIUM

Figure 17 shows the interaction of B, SiC, and TiB_2 with commercial purity titanium. The general microstructures are similar to those for the interaction with high purity titanium. In Figure 6, it can be seen that B, SiC, and TiB_2 is the order of decreasing reactivity although the relative values of the parabolic rate constants are lower in commercial purity titanium than in the higher purity titanium. This slight decrease in reactivity may be the result of the higher impurity levels of Fe, Mn, and W in the commercial purity material.

Figure 18 shows the progressive growth of the reaction layer between B and commercial purity titanium formed at 900°C . After one hour, voids start to form in the boron filament and adjacent to the filament core. In nine hours, substantial void formation is present and the reaction zone has continued to grow. After 100 hours, it can be seen that the reaction zone is separated from the filament at the filament- TiB_2 reaction zone interface. This separation probably occurs on cooling after the reaction layer has grown to a certain thickness. This separation probably did not occur at temperature because the reaction zone is rather uniform. The reaction layer does not separate after only a nine hour anneal at 900°C . This probably indicates the need for the reaction layer to be a certain thickness before separation. In Figure 17 (a), it can be seen that some cracking has been initiated in the TiB_2 phase formed at 850°C although the gross separation is not present.

3. TITANIUM ALLOY: TI-6Al-4V

Figure 19 shows the interaction of B, SiC, and TiB_2 filaments with Ti-6Al-4V titanium alloy after 100 hours at 850°C . Figure 6 shows that B, SiC, and TiB_2 is the order of decreasing reactivity of the filaments in Ti-6Al-4V. High purity, commercial purity, and alloy titanium is of the order of decreasing reactivity with all the filaments.

The reaction of boron with Ti-6Al-4V is slower than with either of the unalloyed titanium metals. Ti-6Al-4V is a two-phase alloy composed of approximately 70% alpha and the remaining beta (Reference 8). Figure 20 shows the white α -phase which completely surrounds the reaction layer. This is probably the result of the formation of the titanium diboride and the rejection of aluminum ahead of the reaction front which stabilizes the α -phase. This may partially explain the reduced rate of reaction of boron with the titanium alloy compared to the two unalloyed titanium matrices.

The SiC and TiB_2 filaments react with Ti-6Al-4V in a way similar to that in the unalloyed titanium. With SiC in Ti-6Al-4V, the reaction zone is multi-layered and both alpha and beta phases are in contact with this reaction zone. The reaction zone between the TiB_2 filament and Ti-6Al-4V is single-layered and is probably the TiB phase.

Parabolic rate constants for the growth of the reaction layer between SiC and Ti-6Al-4V were established at 800, 900, and 991°C . These rate constants were then plotted (Figure 21) to the best fit of the Arrhenius equation

$$k = A e^{-\frac{E}{RT}} \quad (2)$$

where k is the parabolic rate constant, E is the apparent activation energy, R is 1.987 cal/deg-mole, A is a constant, and T is the temperature in degrees Kelvin. The apparent activation energy is 31.3 k cal/mole. By extrapolating this curve to 1200°F (the maximum useful temperature of the titanium alloys), the reaction zone thickness may be calculated using Equation 1. For example at 1200°F for 100 hours, the reaction zone thickness between SiC and Ti-6Al-4V would be approximately 1.5μ .

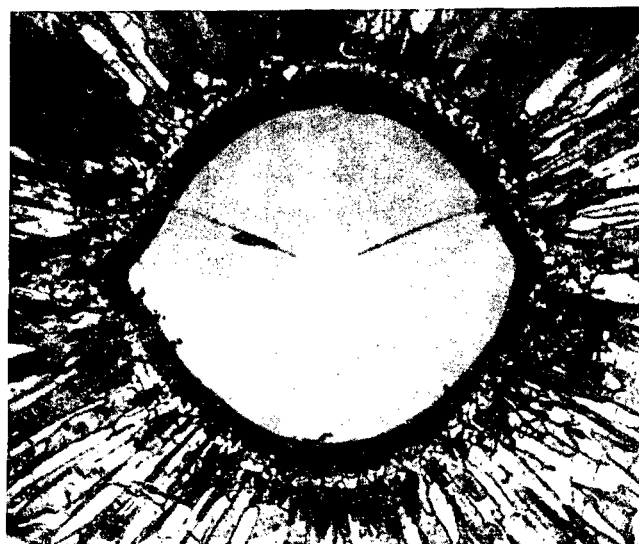
SECTION IV

CONCLUSIONS

1. The order of decreasing filament-matrix interaction is B, SiC, and TiB_2 in the three titanium matrices after 100 hour anneals at 850 to 900°C.
2. High purity, commercial purity, and Ti-6Al-4V alloy titanium is the order of decreasing filament-matrix interaction with the three filaments used in this investigation.
3. The boron filament reacts similarly in both the unalloyed titanium matrices. A coherent TiB_2 layer was formed in contact with the filament after 100 hours at 850°C. A TiB acicular structure was formed adjacent to the TiB_2 phase in the titanium. The boron-titanium interaction takes place by a predominantly one-way diffusion of boron outward which is evident by the lack of recession of the initial boron-metal interface and the void formation in the filaments.
4. The SiC filament reacted with the unalloyed titanium to form a multi-layered reaction zone which was formed by the simultaneous growth outward and inward of the reaction zone. A uniform distribution of approximately 0.1 weight percent titanium was observed in the SiC filament after 100 hours at 850°C. The activation energy for the interaction with Ti-6Al-4V was determined to be 31.3 k cal/mole.
5. The TiB_2 filament reacted with the titanium matrices to form a layer of TiB adjacent to the filament.
6. The reaction of boron with Ti-6Al-4V can be characterized as the formation of TiB_2 with rejection of aluminum ahead of the reaction front which stabilizes the α -titanium phase in contact with the TiB_2 phase.
7. The SiC and TiB_2 filaments do not significantly react with their respective cores after 100 hours at 900°C. The tungsten core of the boron filament appears to be completely converted to the respective tungsten borides during the filament deposition process.

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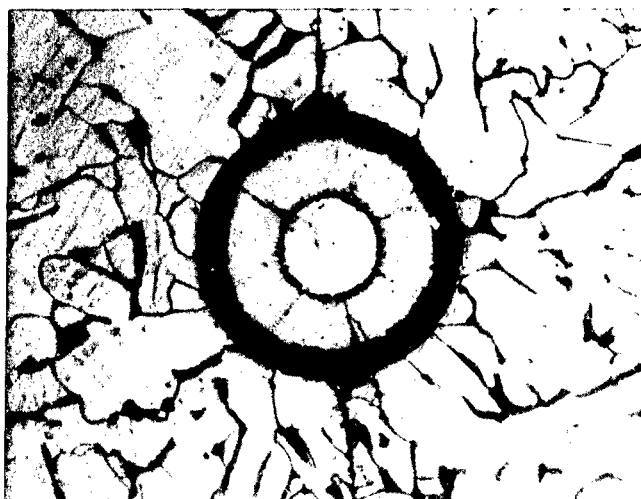
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(a) B, 1 Hour



(b) SiC, 1 Hour



(c) TiB₂, 100 Hours

Figure 1. Comparison of the Reaction of Various Filaments With Iron After a 900°C Anneal for Various Times (550X).



(a) B, 1 Hour



(b) SiC, 1 Hour

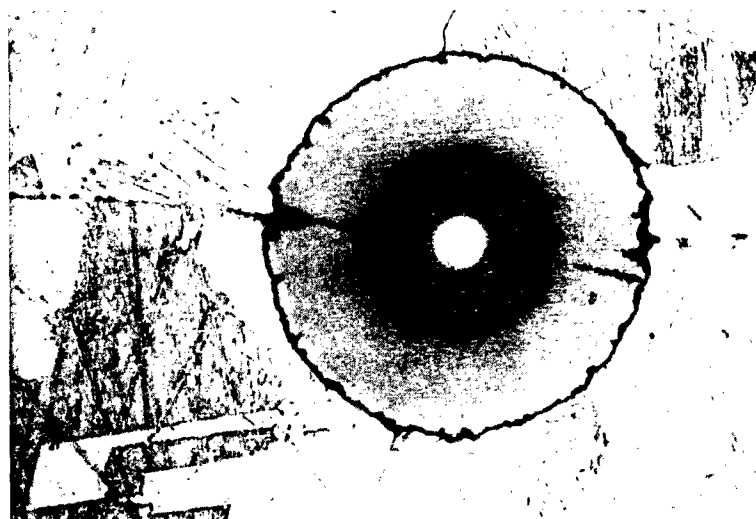


(c) TiB₂, 100 Hours

Figure 2. Comparison of the Reaction of Various Filaments With Nickel After a 900°C Anneal for Various Times (550X)



(c) TiB_2 , 100 Hours



(b) SiC , 1 Hour



(a) B, 1 Hour

Figure 3. Comparison of the Reaction of Various Filaments With Cobalt After a 900°C Anneal for Various Times (550X)

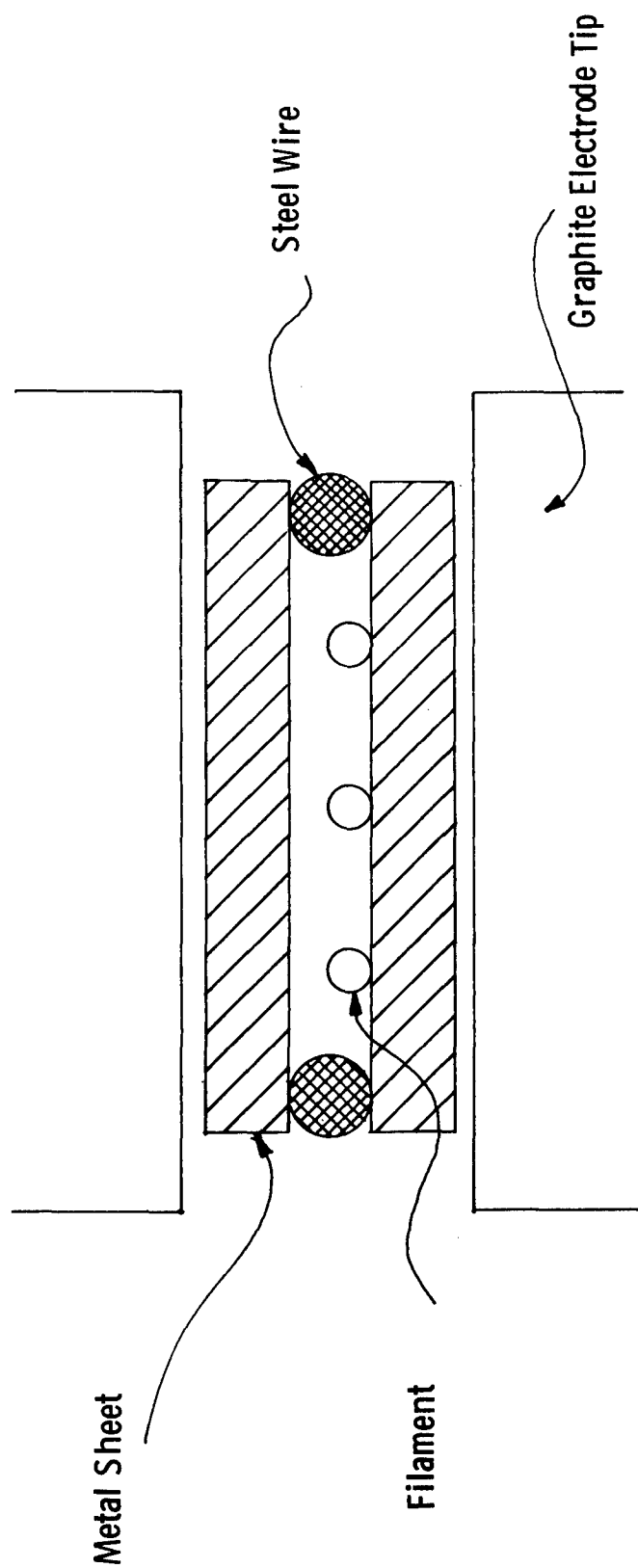
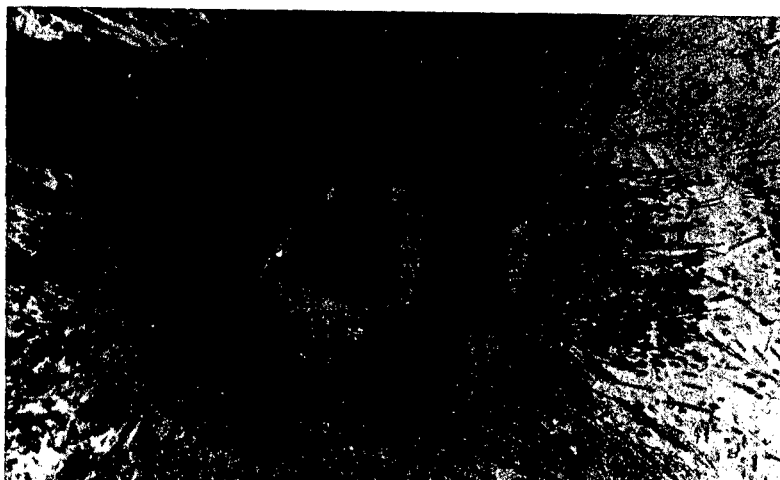
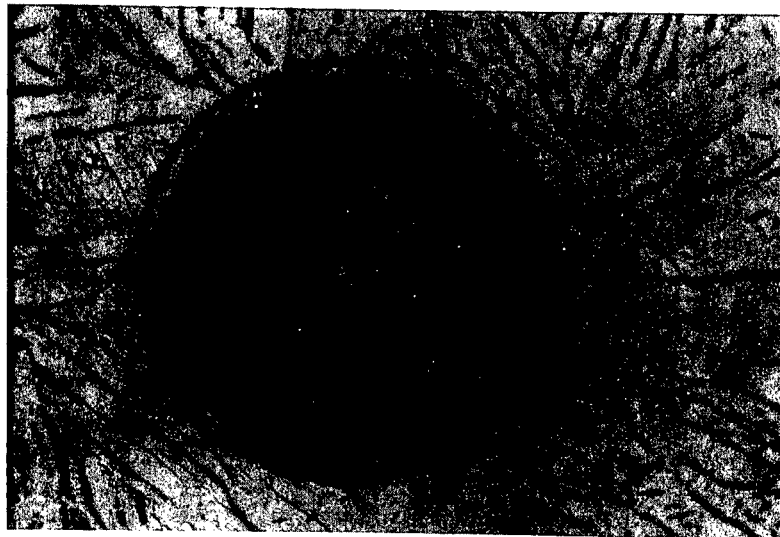


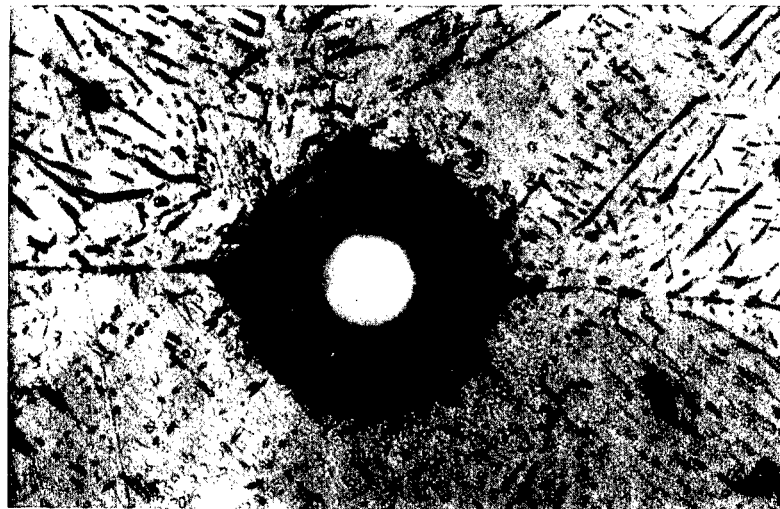
Figure 4. Schematic of Specimen Lay-Up



(a) B Filament



(b) SiC Filament



(c) TiB_2 Filament

Figure 5. Filament-Matrix Interaction in High-Purity Titanium Matrix After a 100 Hour Anneal at 850°C (500X).

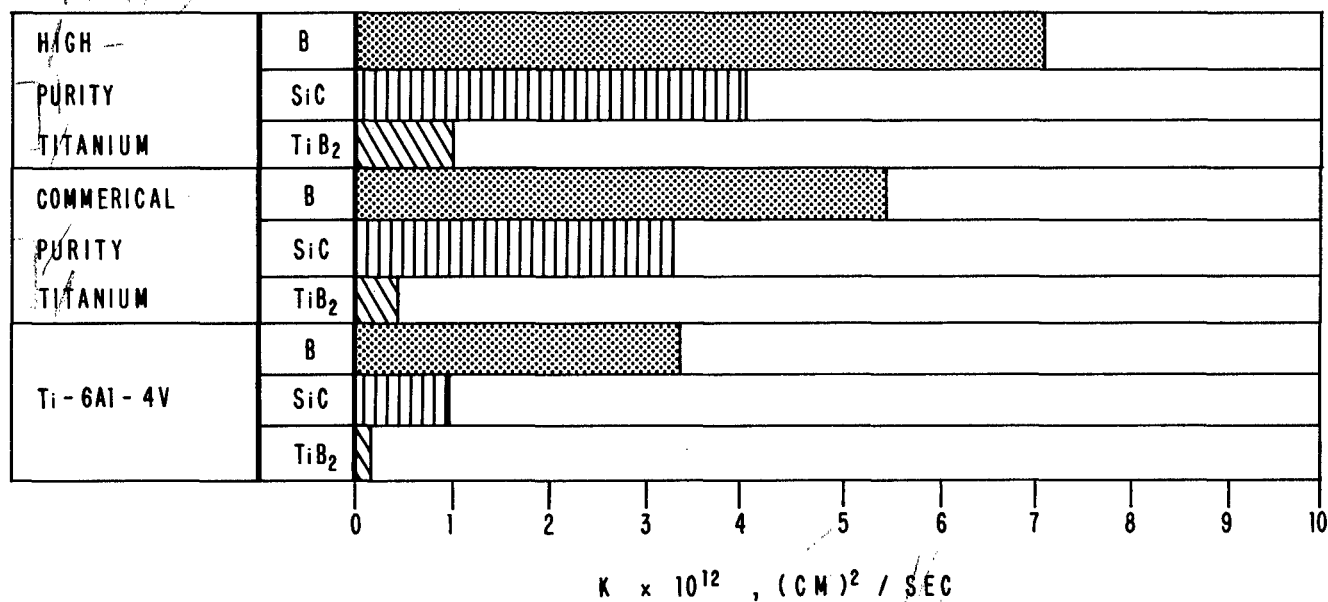


Figure 6. Comparison of the Parabolic Rate Constants for the Filament-Matrix Interaction After 100 Hours at 850°C

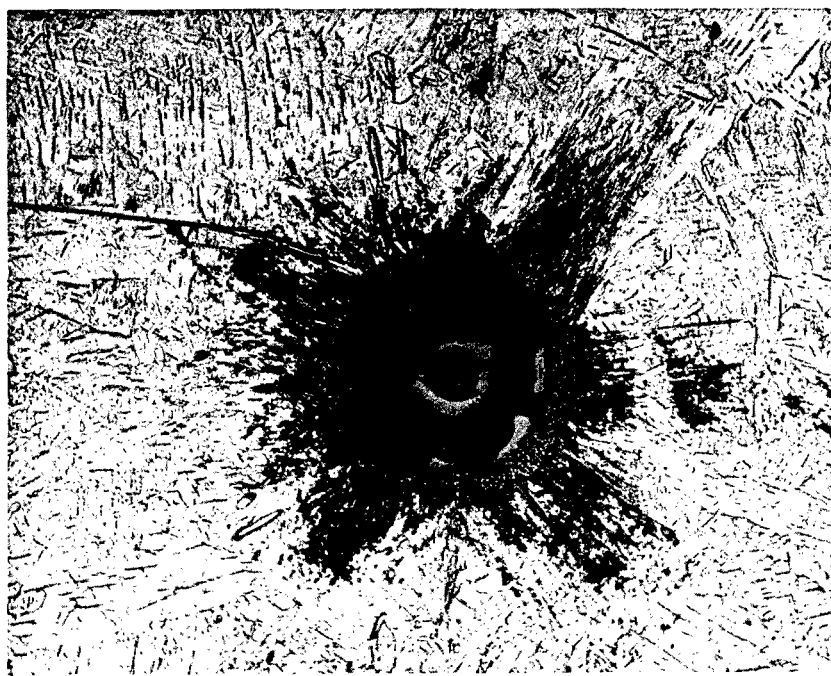


Figure 7. Boron Filament Interaction With High-Purity Titanium After a 100 Hour Anneal at 850°C (250X)

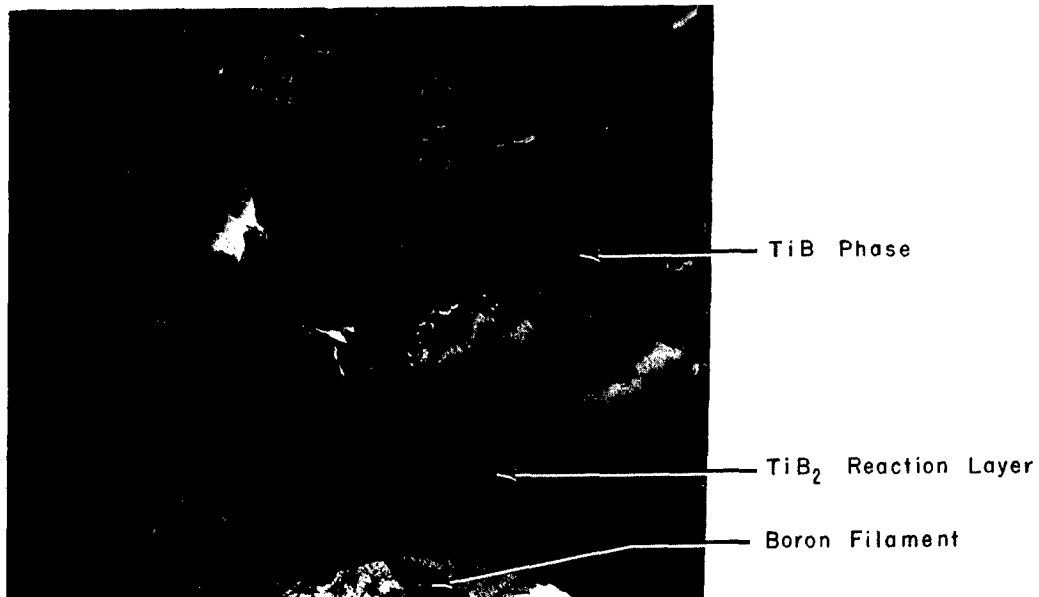


Figure 8. Electron Micrograph of the Reaction Layer Between B and High-Purity Titanium After a 100 Hour Anneal at 850°C (2400X).



Figure 9. Electron Micrograph of the Core of the Boron Filament After a 100 Hour Anneal at 850°C (2400X).

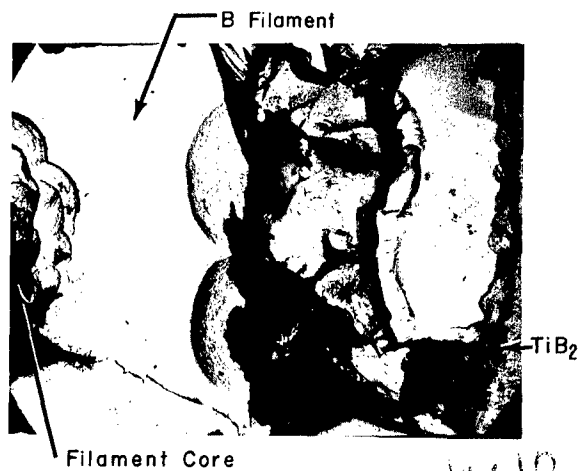


Figure 10. Electron Micrograph of the Boron Filament After 100 Hours at 850°C (1100X).



Figure 11. Silicon-Carbide Interaction With High-Purity Titanium After a 100 Hour Anneal at 850°C (500X).

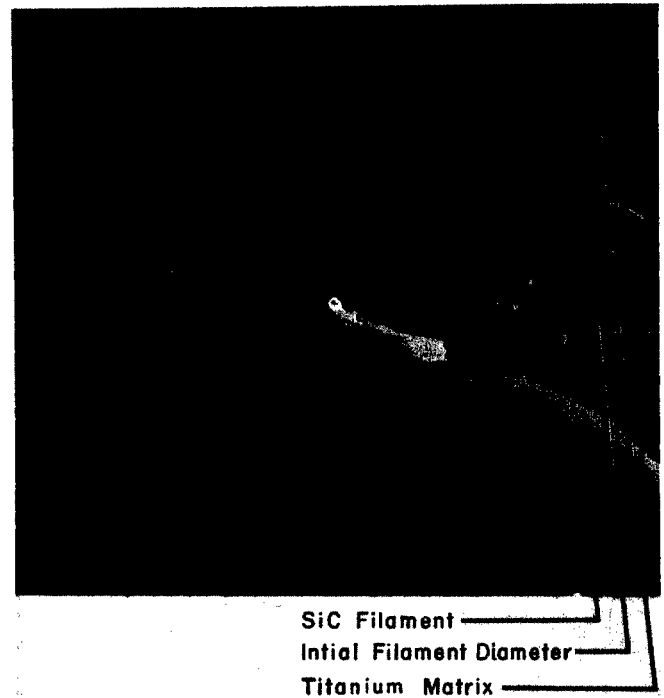


Figure 12. Electron Micrograph of the Reaction Layer Between SiC and High-Purity Titanium After a 100 Hour Anneal at 850°C (2400X).

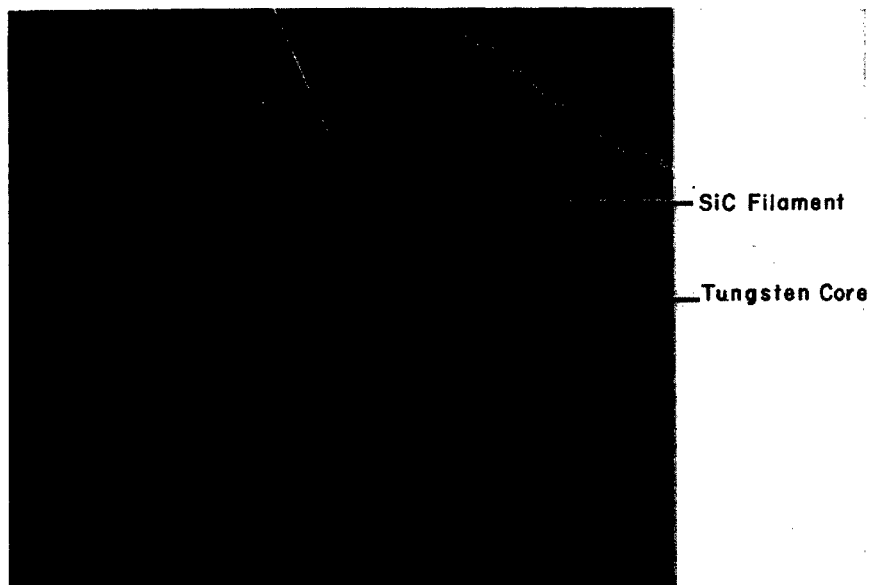


Figure 13. Electron Micrograph of the Tungsten Core of SiC Filament After a 100 Hour Anneal at 850°C (2400X).



Figure 14. Titanium-Diboride Filament Interaction With High-Purity Titanium After a 100 Hour Anneal at 850°C (500X).

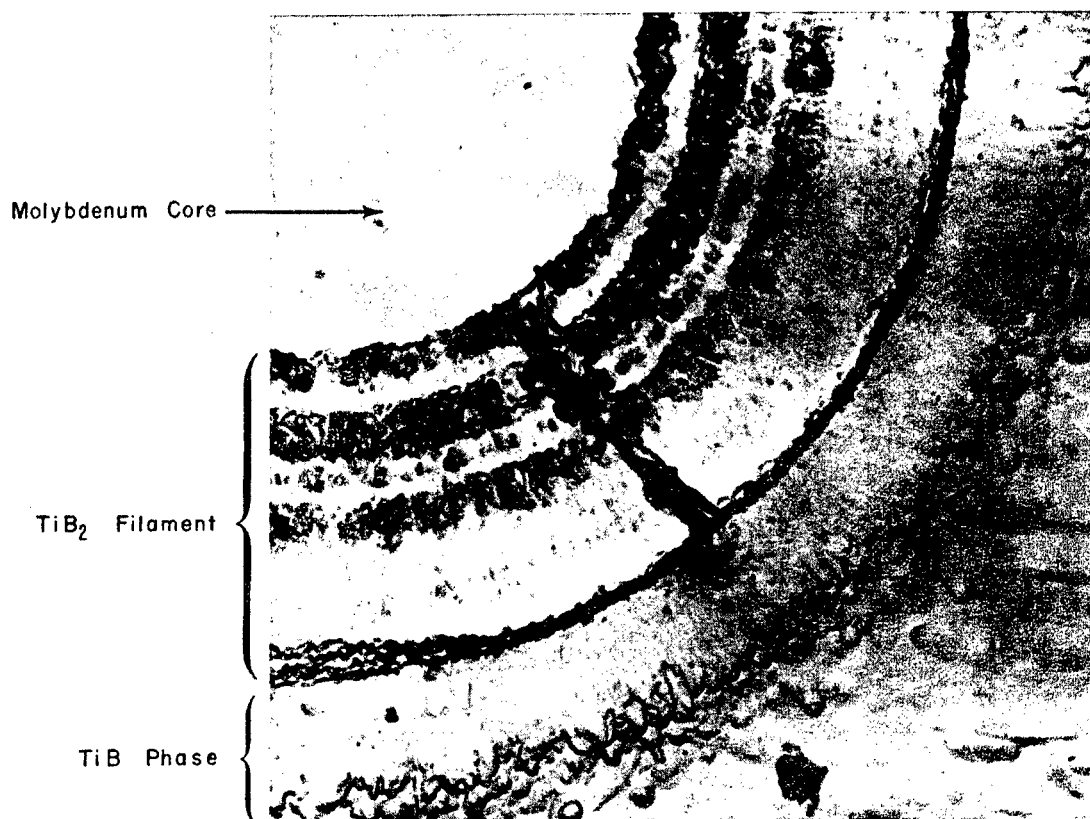
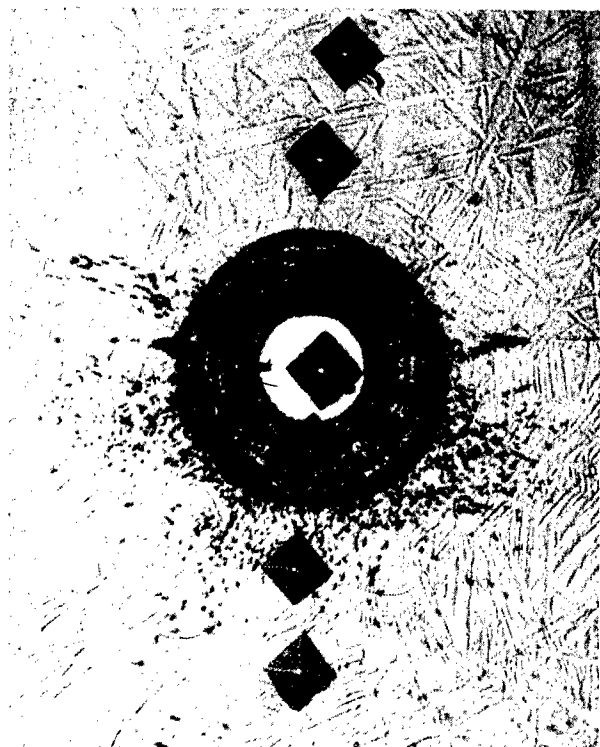
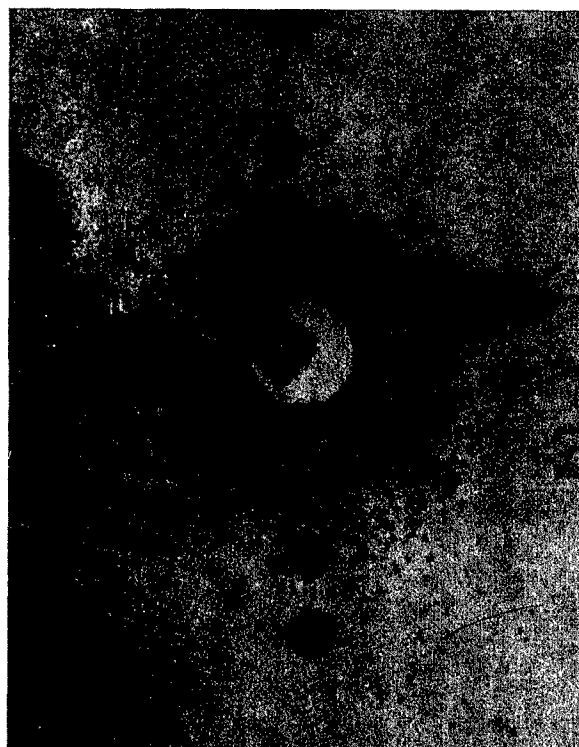


Figure 15. Electron Micrograph of the Reaction Layer Between a TiB₂ Filament With a High-Purity Titanium After a 100 Hour Anneal at 850°C (3200X).



(a) 9 Hours

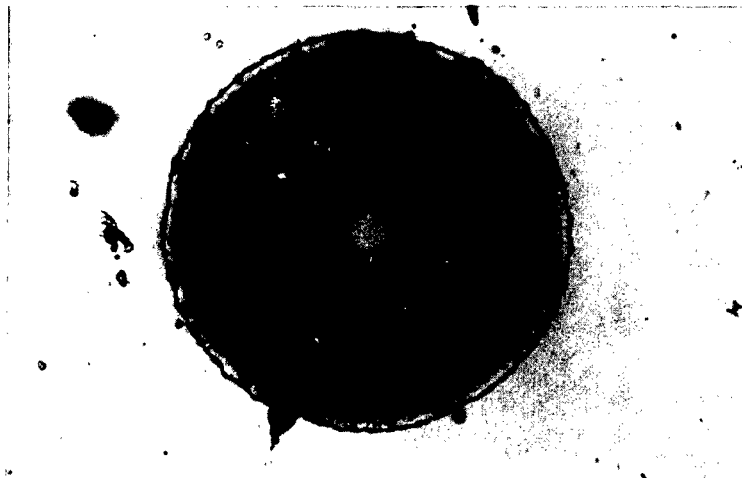


(b) 100 Hours

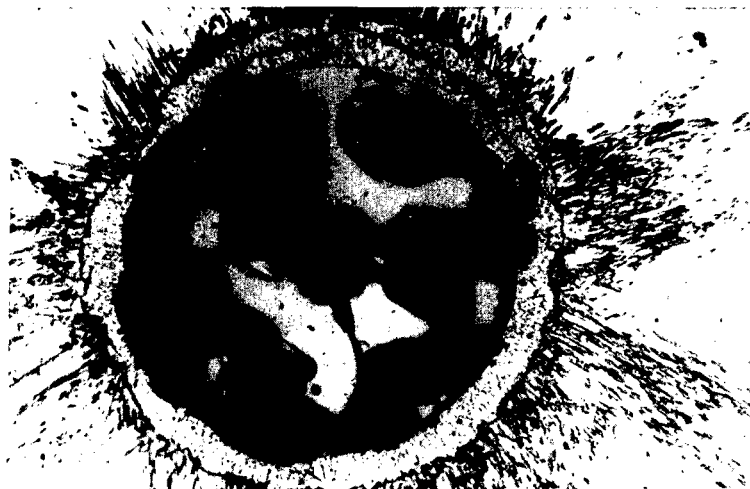
Figure 16. Microhardness Traces Which Show the Increase in Hardness Due to the Filament-Matrix Interactions at 900°C (50 Gram Load - 550X).



(c) TiB_2 Filament

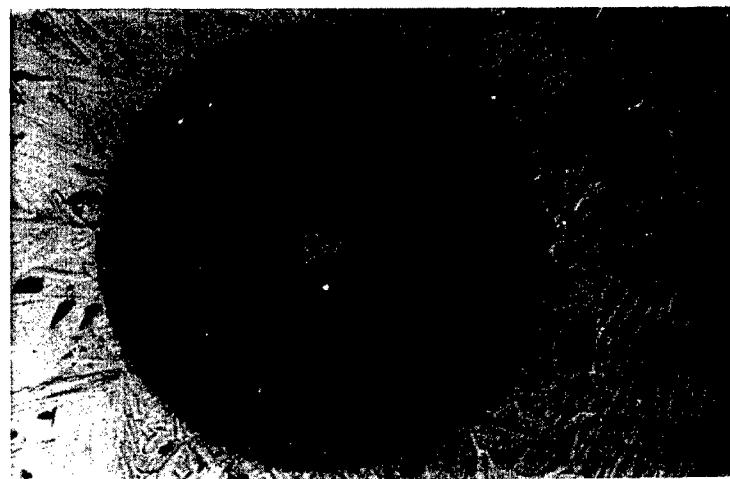


(b) SiC Filament

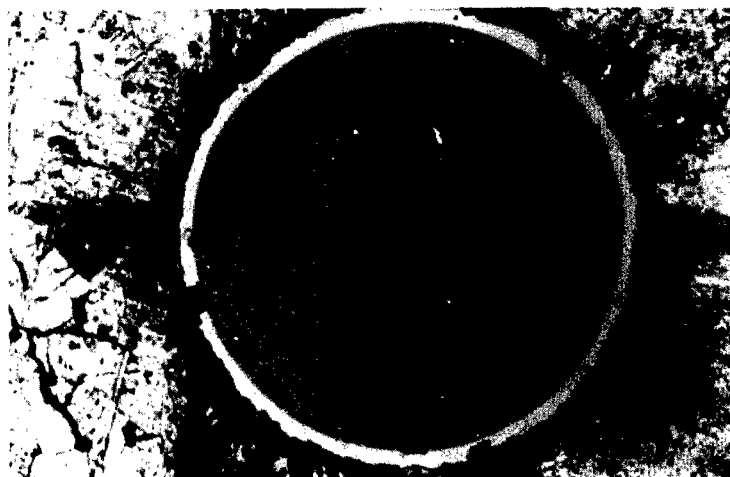


(a) B Filament

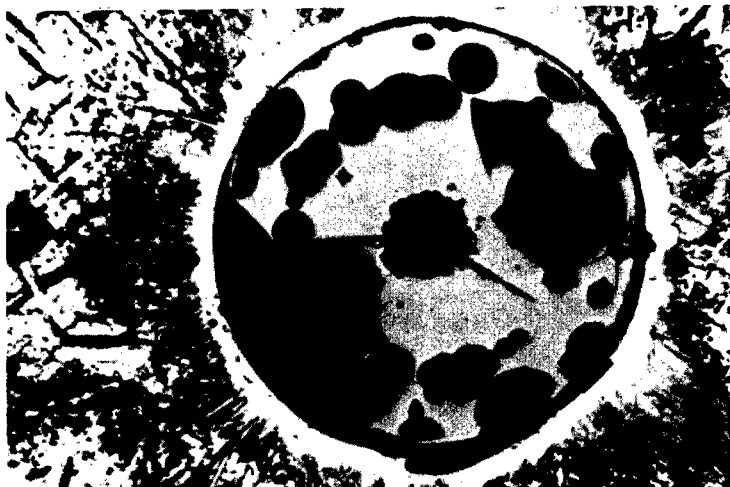
Figure 17. Filament-Matrix Interaction in Commercial Purity Titanium After a 100 Hour Anneal at 850°C (500X).



(a) 1 Hour



(b) 9 Hours



(c) 100 Hours

Figure 18. Isothermal Growth of the Reaction Layer Between the Boron Filament and Commercial Purity Titanium at 900°C (550X).

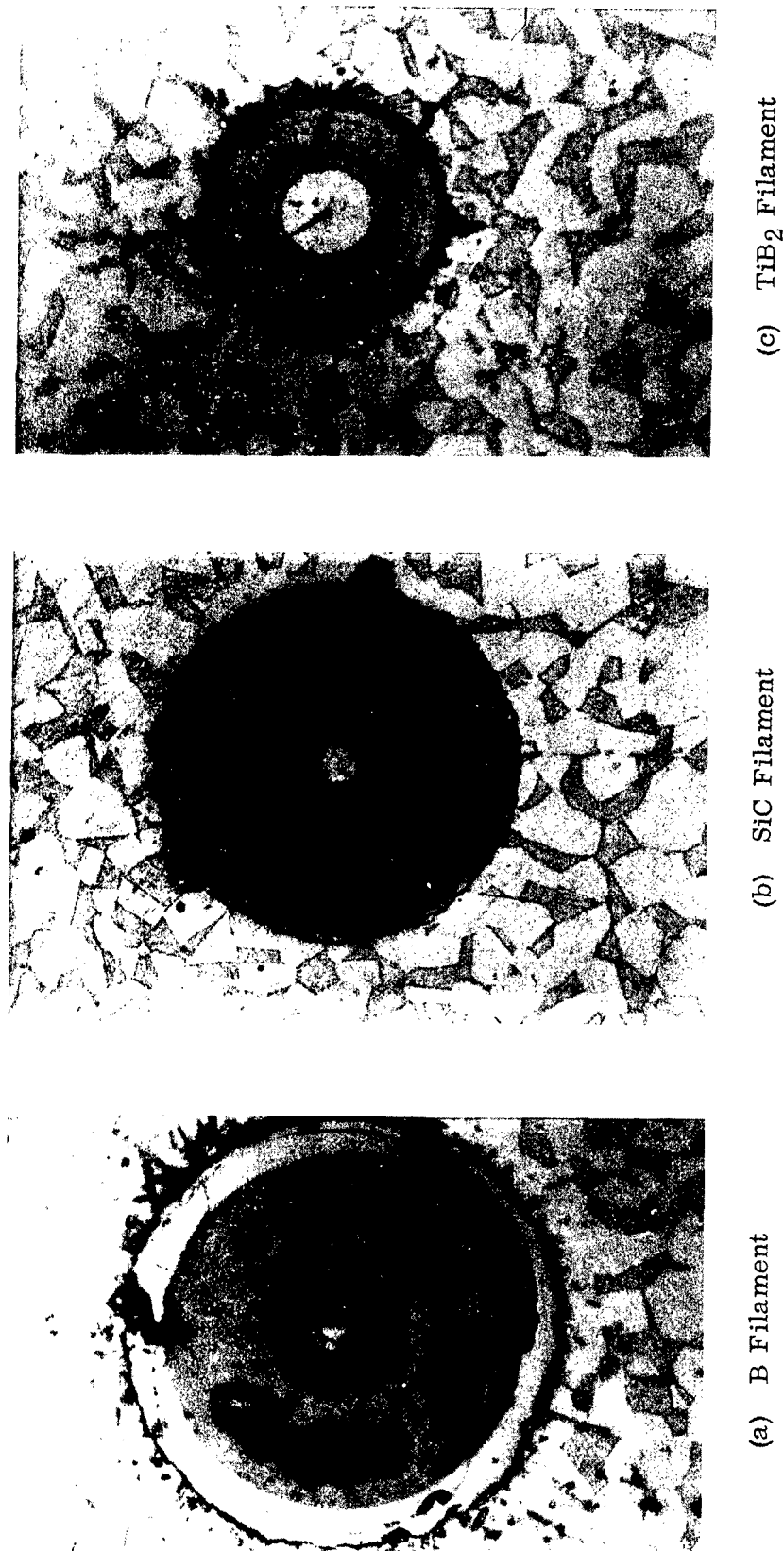


Figure 19. Filament-Matrix Interaction in a Ti-6Al-4V Titanium Alloy 100 Hour Anneal at 850°C (500X).

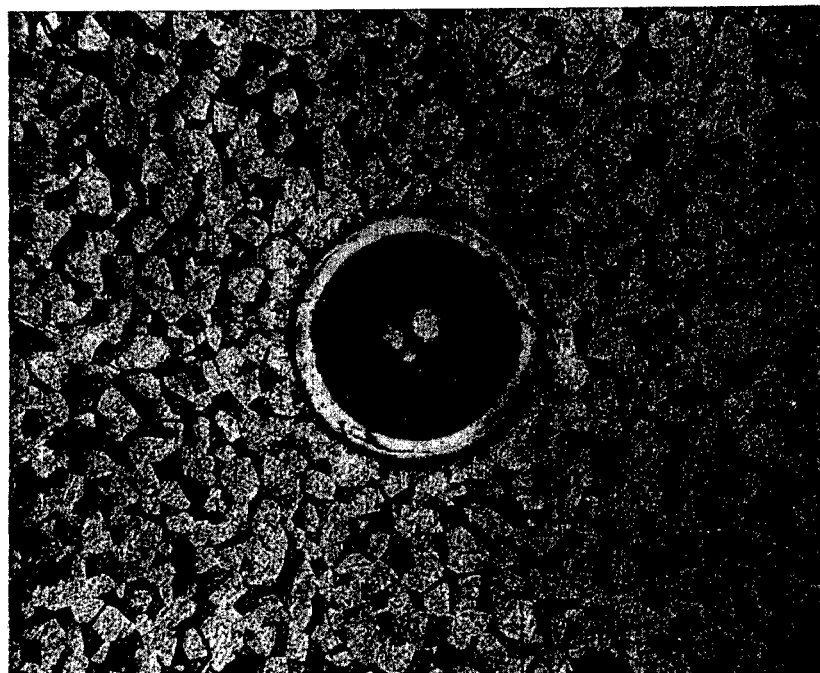


Figure 20. Filament-Matrix Interaction of the Boron Filament With Ti-6Al-4V Titanium Alloy After a 100 Hour Anneal at 850°C (250X).

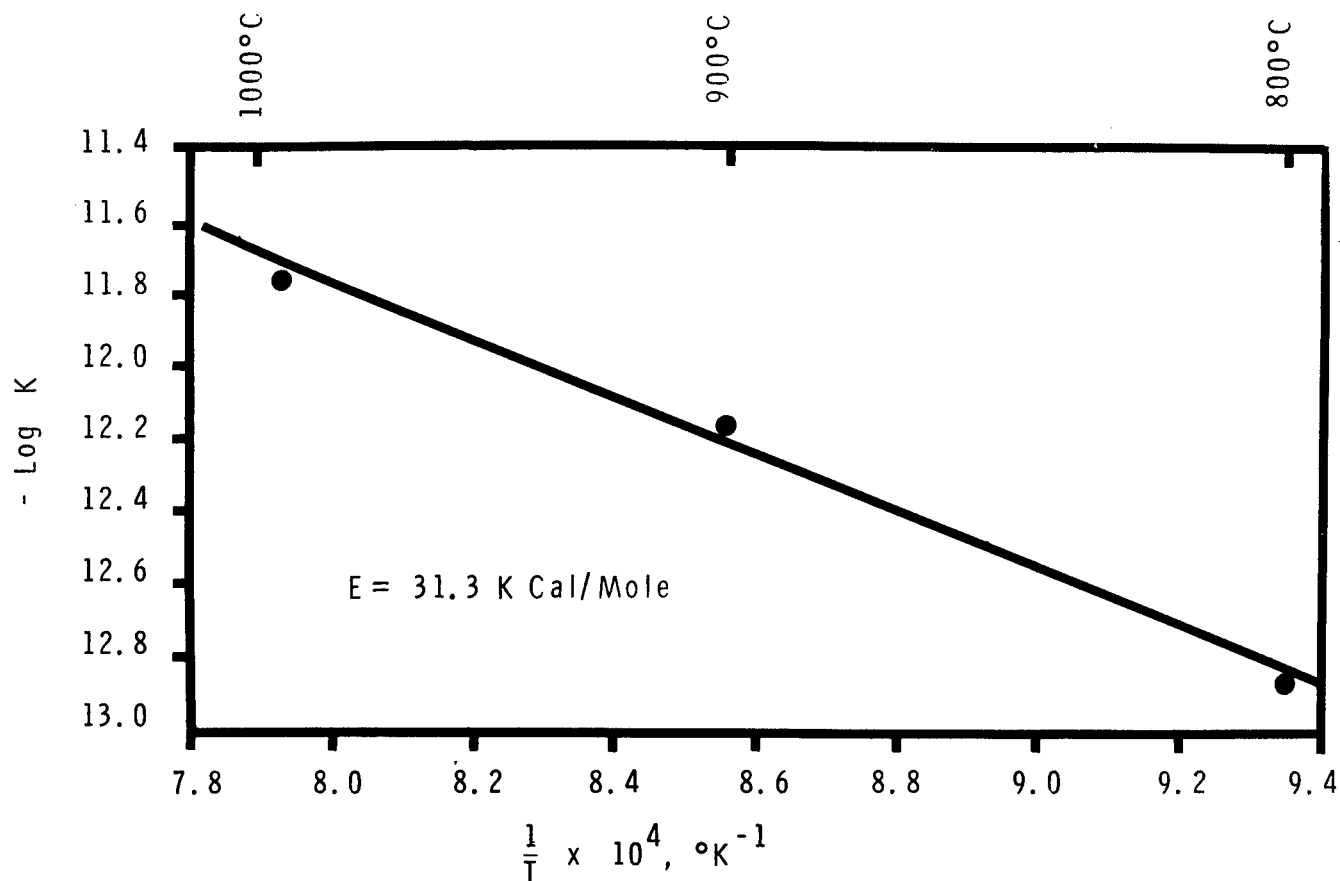


Figure 21. Arrhenius Plot of the Reaction of a SiC Filament With Ti-6Al-4V

TABLE I
FILAMENT DATA

| Filaments | Diameter Mils (μ) | Supplier | σ_{avg} (ksi) | E_{avg} (10^6 psi) | ρ (gm/cm ³) |
|------------------|----------------------------|----------|-------------------------|----------------------------|---------------------------------|
| B | 4.1 (104) | Texaco | 450 | 55 | 2.37 |
| SiC | 3.6 (92) | GTC | 360 | 61 | 3.5 |
| TiB ₂ | 2.6 (66) | GTC | 140 | 65 | 5.2 |

TABLE II
SPECTROGRAPHIC ANALYSIS OF TITANIUM SHEET

| Impurities | Iodide Titanium (PPM) | Commercial Titanium (PPM) | Alloy Ti-6Al-4V (PPM) |
|------------|-----------------------------|---------------------------------|-----------------------------|
| Al | 140 | 170 | (5.9%*) |
| V | ND | ND | (3.8%*) |
| Mg | 20 | 20 | 20 |
| Mn | 140 | 840 | 250 |
| Sn | 20 | 170 | 35 |
| Pb | 20 | 20 | 20 |
| Fe | 20 | 1700 | 1700 |
| Ni | 20 | 80 | 20 |
| Cu | 90 | 170 | 70 |
| Na | ND | ND | 1700 |
| Cr | 20 | 170 | 20 |
| W | 20 | 330 | 200 |
| Ca | 170 | 170 | 200 |
| Mo | 20 | 20 | 200 |

* Atomic Absorption

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13. ABSTRACT

The filament-matrix compatibility was evaluated for B, SiC, and TiB₂ vapor deposited filaments with high purity, commercial purity titanium, and Ti-6Al-4V. Filament-matrix diffusion sandwiches were prepared by solid state resistance bonding. These samples were subsequently heated at 800-991°C for times of 1-100 hours. The reaction rates and reaction products were evaluated using light microscopy, electron replicas, microhardness measurements, and microprobe analysis.

The order of decreasing filament-matrix interaction was B, SiC, and TiB₂ in each of the three materials. High purity Ti, commercial purity Ti, and Ti-6Al-4V is the order of decreasing reactivity with the filaments after 100 hours at 850-991°C. The reaction of B with unalloyed titanium is characterized as the formation of a TiB₂ layer adjacent to the filament with an external acicular TiB layer. The reaction layers were formed by the predominately outward diffusion of the boron as evidenced by the lack of recession of the initial filament diameter and the void formation in the filament. The interaction of B with Ti-6Al-4V is characterized by the formation of TiB₂ with the rejection of Al ahead of the advancing TiB₂ front which stabilizes the α -titanium phase around the TiB₂ reaction layer. The SiC filament reacts with titanium to form a multi-phased reaction layer. The SiC-Ti interaction takes place by the simultaneous growth inward and outward of the reaction zone. A uniform distribution of 0.1 weight percent titanium was observed in the SiC filament after 100 hours at 850°C. The TiB₂ filament reacted with titanium to form a TiB layer adjacent to the filament.

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The SiC and TiB₂ filaments did not react with their respective tungsten or molybdenum core after 100 hours at 850°C.

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